

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Patent Application of)	
Catherine Hedouin)	Group Art Unit: 1793
Application No.: 10/519,040)	Examiner: Diana J. Liao
Filed: September 21, 2005)	Confirmation No.: 7039
For: COMPOSITION BASED ON)	
ZIRCONIUM OXIDE AND OXIDES)	
OF CERIUM, LANTHANUM AND OF)	
ANOTHER RARE EARTH, A)	
METHOD FOR PREPARING SAME)	
AND USE THEREOF AS)	
CATALYST)	

DECLARATION PURSUANT TO 37 C.F.R. §1.132 OF SIMON IFRAH, PhD

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Sir:

1. I, Simon Ifrah, declare the following:
2. I, Simon Ifrah, am a citizen of France.
3. I received a PhD from the University of Lyon for my studies in the field

of chemistry.

4. I have been employed by Rhodia from 2006 to the present. I am currently engaged in research and development in the field of rare earth mineral synthesis.

5. I am familiar with the subject matter of the above identified U.S. patent application, including the content of claim 17 currently contained therein:

17. A composition based on zirconium oxide comprising cerium oxide in an atomic ratio Zr/Ce > 1, and in addition comprising lanthanum oxide and an oxide of a rare earth other than cerium and lanthanum, the composition having a sulfur content below 200 ppm, wherein after

calcination for 6 hours at 1150°C it has a specific surface of at least 10 m²/g.

6. I have reviewed international patent application publication no. WO 95/35152 (hereafter WO '152) cited against the above claim by the U.S. Patent Office.

7. I understand that it has been alleged by the U.S. Patent Office that the surface area stability property recited in the above claim ("*after calcination for 6 hours at 1150°C it has a specific surface of at least 10 m²/g*") is inherent and obvious to the composition described by WO '152.

8. Based on the experiments described hereafter, I do not agree that the above-quoted property is either inherent to or obvious in view of the WO '152 composition.

9. The following experiments are intended to replicate the Example described on page 34 of WO '152 (first layer) as closely as possible to produce a product comparable with the composition recited in claim 17. The following experiments were conducted under my direct supervision.

10. For these experiments the following reagents were used:

- zirconyl chloride solution with a concentration 279 g/l, and a density of 1.361 g/cm³, and a ratio of Cl/ZrO₂ = 2.06;
- cerium nitrate solution with a concentration of 496.4 g/l, and a density 1.716 g/cm³;
- NH₄OH with a concentration = 11.9 N;
- lanthanum nitrate solution with a concentration of 450 g/l;
- neodymium nitrate solution with a concentration of 519 g/l;
- strontium acetate hemihydrate with 98% oxide content (SrO) 50.2%;

- zirconium acetate solution with a concentration of 288 g/l; and
- acetic acid 100%.

11. A ceria-zirconia powder comprising 20 wt% of CeO_2 was prepared by precipitation with ammonia of a mixture of zirconyl chloride and cerium nitrate solutions. The precipitate which was obtained was calcined in a muffle furnace at 470°C for 5 hours. The product which was obtained exhibited a specific surface area of $54.7\text{m}^2/\text{g}$.

12. 972 grams of the above calcined ceria-zirconia powder was combined with lanthanum nitrate in an amount sufficient to form 146 grams La_2O_3 , neodymium nitrate in an amount sufficient to form 194 grams Nd_2O_3 , strontium acetate in an amount sufficient to form 486 grams SrO , zirconium acetate solution in an amount sufficient to form 97 grams ZrO_2 .

13. The slurry which was obtained was ballmilled for 30 minutes at 500r/min in a NETSCH Molinex ball mill (0.6 mm zirconia balls) with 148 grams of glacial acetic acid and sufficient water to form a 48 percent by weight solids aqueous slurry.

14. The resulting slurry was poured into a critallizer and dried for 20 minutes at 100°C in a stove.

15. The dried product was then calcined for 30 minutes at 450°C in a muffle furnace with a rate of temperature rise of $0.5^\circ\text{C}/\text{min}$.

16. The resulting product presented a specific surface area of $15.6\text{m}^2/\text{g}$.

17. The resulting product was then tested for high-temperature surface area stability by heating (calcining) for 6 hours at 1150°C in a muffle furnace with a rate of temperature rise of $1^\circ\text{C}/\text{min}$.

18. Afterwards, the product exhibited a specific surface area of 1.1 m²/g. This value is far short of the 10 m²/g surface area after heating under these conditions, as required by claim 17.

19. I further declare that all statements made herein of my own knowledge are true and that all statements on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful statements may jeopardize the validity of the application or any patent issuing thereon.

Date: 05/11/2009

By: SIMON IFRAM
Simon Ifrah